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# DISSOLVING ORES AND REFRACTORIES BY SODIUM PEROXIDE FUSION IN A VITREOUS GRAPHITE CRUCIBLE

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ABSTRACT. For the analysis of chromites or refractories, vitreous graphite crucibles 50 mm high, outer diameter 50 mm, inner diameter 45 mm are preferable to Fe, Ni, or Pt crucibles. Fusion with $Na_2O_2$ is more complete, especially for Fe ores. After washing with $H_2O$ and oven drying the graphite crucible can be reused 10 - 15 times. For 3 std. samples recoveries of Fe, Si, Al, and Ti were essentially quant.

## I. INTRODUCTION /1109**

One of the basic ideas of analytical chemistry, though it is rarely stated explicitly, is the following: never undertake an analysis as long as it is not certain that the analytical sample has been completely dissolved.

Also, the chemist constantly runs into many different products which resist attack by acids or other reagents. These include certain refractories, chromite present in laterites, a certain number of rocks, and various inclusions in steels.

After prolonged attacks at ambient temperature, analysts have studied calcinations and fusions with different compounds such as borates, carbonates, and sodium peroxide [1]. Although this often ends in a satisfactory attack

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on the sample with production of an acid-soluble "bead," these techniques involve a large increase in temperature, which then raises the new question of the behavior of the fusion crucible.

For example, it is known that platinum is attacked by alkaline melts at high temperatures. Substantial amounts of platinum pass into solution, leading for example, to high analyses when iron is determined volumetrically with potassium dichromate, while the colorimetry of iron with orthophenantrholine is not disturbed. Inversely, if a nickel crucible is used [4, 5, 6, 7, 8, 9], volumetric analysis of iron is not affected, but colorimetry leads to low values of iron.

The same problems occur with alumina, fused-lime or other crucibles, since determinations of aluminum or calcium in the sample are often desired, /1110 while zirconium from an attack on a zircon or zirconia crucible interferes by the formation of precipitates.

One would thus like to find a material resistant to the media for calcining or high-temperature fusion, whose constituent elements do not pass into solution.

It appears that this product is vitreous graphite, with which the tests described below were carried out.

## II. DESCRIPTION OF VITREOUS-GRAPHITE CRUCIBLES

For our tests we used V 25 crucibles produced by Carbone Lorraine. These crucibles are 50 mm high, with an external diameter of 50 mm, and an internal diameter of 45 mm.

Although they are sensitive to mechanical shocks, they have good resistance to thermal shocks. These crucibles have closed porosity, and are thus impermeable to gases.

The vitreous-graphite crucibles resist hydrochloric, nitric, sulfuric, and hydrofluoric acids, as well as caustic alkalis. This is also true for perchloric acid, but in this case our tests were limited to ambient temperature.

The crucibles are impermeable to these materials, as can be shown by placing them on pH paper for a long time.

## III. EXPERIMENTAL SECTION

Extending the experiment of calcining with sodium peroxide at 400° C, required for disintegration of iron ores in particular, we have tried to pass to fusion of sodium peroxide, or to temperatures of 600 to 800° C.

In a first group of tests, we have tried various fusions and controlled the weight-loss of crucibles weighing about 12 gm. The results are as follows:

| Nature of Test | Duration (min) | Weight loss (gm) |
|---|---|---|
| Vacuum calcination, in air in muffle furnace, 1000° C | 10 | 1.60 |
| $Na_2O_2$ calcination, in air in muffle furnace, 400° C | 60 | 0.21 |
| $NaHSO_4$ fusion, Mecker burner | 5 | 0.03 |
| Fusion of equimolar mixture of $Na_2CO_3$ and $Na_2B_4O_7$, Mecker burner | 20 | 0.32 |
| $LiB_4O_7$ fusion, in air in muffle furnace, 1200° C | 10 | 1.26 |
| $Na_2O_2$ fusion, Mecker burner | Destruction of crucible bottom. | |

When the fusion is carried out with only sodium peroxide in the vitreous-graphite crucible, a very luminous ignition point is observed each time, which leads to perforation of the crucible bottom. At 600 to 800° C, the reaction between the sodium peroxide oxidant and the carbon reductant becomes very easy.

It is noted, however, that with 2.5 gm of alumina refractory and 7 to 8 gm of sodium peroxide this ignition, which always occurs, is much less intense. There is thus a favorable dilution effect.

During the tests, it was seen that for "basic" products such as calcareous ores or magnesia refractories, the fusion was much quieter, often without ignition, unlike the case for acidic products, particularly silicacous ones. This has led us to add 1 gm of sodium carbonate.

Thus, with the fusion method described above, one can disintegrate and then easily dissolve a very large number of products difficult to attack by other methods. This operating mode allows use of a vitreous-graphite crucible for 10 to 15 times before the successive weight losses make it too prone to break.

Operating Mode

Add a 2.5-gm analytical sample to 7 to 8 gm of sodium peroxide and 1 gm of sodium carbonate. Heat gently with a Mecker burner applied to the crucible with a circular movement. After a few minutes, when the mass to be fused is well dehydrated, increase the heating. A viscous melt will be observed. Verify, by observing the color of the crucible bottom, that no preferential heating has occurred.

If such heating has occurred, let the crucible cool a little with constant agitation, placing it on a ceramic plate for an instant if necessary. Then resume heating until a well-melted mass is obtained (5 to 10 minutes).

After cooling, place the crucible and its contents in a 600-ml beaker /1111
covered with a watch glass. Add 250 ml of water, 50 ml of hydrochloric acid

(d = 1.19), and 50 ml of perchloric acid (d = 1.61); proceed with the analyses in the usual way. After rinsing with distilled water and drying, the crucible is ready for another fusion.

Descomps [2, 3] has recently proposed the addition of disodium phosphate. We prefer sodium carbonate, since it is thereby possible to determine a large number of elements after a single fusion, including phosphorus.

There are several advantages to using vitreous-graphite crucibles. One is the absence of any contamination by the crucible, which could falsify or interfere with later determinations. Another is the easy quantitative recovery of the analytical sample, as shown by use of IRSID samples of iron conglomerates and ores AO 1-1, AO 2-1, MO 1-1, MO 2-1, MO 3-1, MO 4-1, MO 5-1, and MO 6-1 and American refractory samples:

| No. | | Fe | $SiO_2$ | $Al_2O_3$ | $TiO_2$ | $Cr_2O_3$ | MgO |
|---|---|---|---|---|---|---|---|
| 76 | certified | 1.66 | 54.68 | 37.70 | 2.21 | - | - |
| | found | 1.57 | 54.65 | 38.00 | 2.15 | - | - |
| 77 | certified | 0.63 | 32.38 | 59.39 | 2.93 | - | - |
| | found | 0.67 | 32.20 | 59.30 | 3.07 | - | - |
| 78 | certified | 0.55 | 20.69 | 69.97 | 3.37 | - | - |
| | found | 0.57 | 21.05 | 69.60 | 3.40 | - | - |
| 103A | certified | - | 4.63 | 29.96 | - | 32.06 | 18.54 |
| | found | - | 4.69 | 29.75 | - | 31.65 | 18.31 |

Note: Safety glasses must be worn at all times when sodium peroxide is used.

## IV. CONCLUSIONS

It has been established that the use of vitreous-graphite crucibles eliminates all interferences and contaminations previously encountered with the use of iron, nickel, or platinum crucibles. Because of the presence of sodium peroxide and a high temperature, a very large number of materials reputed to be difficult to attack can be dissolved rapidly and completely in 15 to 20 minutes without double fusion, and even trace analysis can be performed. Furthermore, the presence of lead, arsenic, or tin does not interfere, as it would with platinum, for example.

These crucibles have no open porosities, and so they can be reused 10 to 15 times without risk of contamination.

## REFERENCES


1. Belcher, C. B. Talanta, Vol. 10, No. 1, 1963, pp. 75-81.
2. Descomps. Information Chimie, Vol. 81, February, 1970, p. 110.
3. Descomps. Chimie analytique, Vol. 50, No. 3, 1968, pp. 54-55.
4. AFNOR, French Standard NF B49-410.
5. Voinovitch, I. A. et al. L'Analyse des Silicates (Analysis of Silicates). Hermann, pub., 1962.
6. Grillot, H. et al. Methodes d'Analyse Quantitative Appliquees aux Roches at aux Prélèvements de la Prospection Géochimique (Methods of Quantitative Analysis Applied to Rocks and to Samples from Geochemical Prospecting), Editions BRGM, No. 30, 1964.
7. Jourdan, A. La Technologie des Produits Céramiques Réfractaires (Technology of Refractory Ceramic Products), Gauthier-Villars, pub.
8. Bennett, H. et al. Trans. Br. Ceram. Soc., Vol. 65, No. 12, 1966, pp. 681-692.
9. Riley, S. P. Anal. Chim. Acta., Vol. 19, 1958, pp. 413-428.

10. Biskupsky, V. S. Anal. Chim. Acta, Vol. 33, 1965, pp. 333-334.

11. Schaffer, H. N. S. Analyst, Vol. 91, 1966, pp. 755-770.

12. Ingamelis, C. O. Anal. Chem., 1966, pp. 1228-1234.

13. Arhens, L. H. et al. Anal. Chim. Acta, Vol. 28, 1963, pp. 551-573.

14. Kics, E. Anal. Chim. Acta., Vol. 39, No. 2, 1967, pp. 223-234.

15. Dolezal, S. et al. Decomposition Technique (Technical Decomposition). In: Inorganic Analysis, ILIFFE Books, London.

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